	Type	L #	Hits	Search Text	DBs	Time Stamp
1	IS&R	L1	1	("6443166").PN.	USPAT	2003/01/02 08:50
2	IS&R	L2	2	(("5168709") or ("5813849")).PN.	USPAT	2003/01/02 09:38
3	BRS	L3	126	55/428.1,467.1.ccls.	USPAT	2003/01/02 09:38
4	BRS	L4	1	3 and chlorine	USPAT	2003/01/02 09:43
5	BRS	L5	1	3 and (sulfur adj dioxide)	USPAT	2003/01/02 09:42
6	BRS	L6	0	2 and (sulfur adj dioxide)	USPAT	2003/01/02 09:43
7	BRS	L7	0	2 and chlorine	USPAT	2003/01/02 09:43
8	BRS	L8	0	2 and nitrogen	USPAT	2003/01/02 09:44
9	BRS	L9	1482	134/22.1,22.16,22.17,22.18, 22.19.ccls.	USPAT	2003/01/02 09:45
10	BRS	L10	106	9 and chlorine	USPAT	2003/01/02 09:46
11	BRS	L11	15	10 and (sulfur)	USPAT	2003/01/02 10:03
12	BRS	L12	3	10 and (sulfur adj dioxide)	USPAT	2003/01/02 10:09
13	BRS	L13	1105	134/10,11.ccls.	USPAT	2003/01/02 10:09
14	BRS	L15	91	13 and chlorine	USPAT	2003/01/02 10:10
15	BRS	L16	91	15 and 15	USPAT	2003/01/02 10:10
16	BRS	L17	3	15 and 14	USPAT	2003/01/02 10:10
17	BRS	L14	9	13 and (sulfur adj dioxide)	USPAT	2003/01/02 10:13

US-PAT-NO: **6443166**
DOCUMENT-IDENTIFIER: US 6443166 B1

TITLE: Method of cleaning a pressurized container

DATE-ISSUED: September 3, 2002

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP	CODE	COUNTRY
Tunney; Joseph P.	Evanston	IL	N/A		N/A
Buchan; Paul	Regina Saskatchewan	N/A	N/A		CA

US-CL-CURRENT: 134/22.1; 134/22.18

CLAIMS:

We claim:

1. A method of cleaning a container having at least one chemical therein, the method comprising the steps of: providing a container having an amount of at least one chemical contained therein wherein the container has a plurality of valves for adding material to the container or removing material from the container; adding an amount of heated nitrogen gas to the container to mix with the chemical creating a nitrogen gas/chemical mixture; providing a flare to incinerate the nitrogen gas/chemical mixture; and venting the nitrogen gas/chemical mixture to the flare.

2. The method of claim 1 wherein the chemical is in a gaseous state at standard temperature and pressure and further wherein the chemical has a pressure within the container.

3. The method of claim 2 further comprising the steps of:

measuring the pressure
within the container prior to adding the heated nitrogen gas; and venting the
nitrogen gas/chemical mixture to the flare.

4. The method of claim 1 further comprising the step of: inspecting the container
for leaks.

5. The method of claim 1 further comprising the step of: sampling the hazardous
material to verify the identity of the chemical.

6. The method of claim 1 further comprising the step of: attaching a pipe to one of
the valves and connecting the pipe to the flare prior to venting the residual
pressure of the chemical.

7. The method of claim 1 further comprising the steps of: providing a housing
having sideports wherein the housing is disposed over the valves; and sampling the
gas within the housing for leaks via the sideports of the housing while inspecting
the container.

8. The method of claim 7 further comprising the steps of: providing a lid on the
housing; opening the lid; and inspecting the valves in the housing for damage to
the valves.

9. The method of claim 1 wherein the temperature of the heated nitrogen gas is
between about 100.degree. F. and about 300.degree. F.

10. The method of claim 1 further comprising the step of: attaching a pipe from the
nitrogen gas to the inlet valve of the container.

11. The method of claim 1 further comprising the steps of: filling the container
with heated nitrogen gas; venting the heated nitrogen gas from the container; and
repeating the filling and the venting a plurality of times until the concentration
of the chemical therein reaches a predefined amount.

12. The method of claim 1 further comprising the step of: adding heated nitrogen gas until the pressure within the container is between about 5 psi and about 50 psi.

13. The method of claim 1 further comprising the step of: grounding the container prior to inspecting the container.

14. The method of claim 1 further comprising the steps of: providing a means to access the inside of the container; and opening the means to access the inside of the container to gain access to the inside of the container.

15. The method of claim 1 further comprising the steps of: providing steam in a steam tank; connecting a pipe from the steam tank to the inlet valve of the container; adding the steam to the container to pressurize the container with the steam; and maintaining the pressure of the steam within the container for an amount of time.

16. The method of claim 1 further comprising the steps of: measuring a residual pressure of the chemical within the container with a pressure gauge prior to adding the amount of heated nitrogen gas; and venting the residual pressure of the hazardous material to the flare.

17. The method of claim 16 further comprising the step of: adding a quantity of heated nitrogen gas if the residual pressure within the container is about zero psi.

18. The method of claim 1 wherein the container is a rail tank car.

19. The method of claim 1 wherein the container has a plurality of chemicals contained therein.

20. The method of claim 1 further comprising the step of:

providing a control panel
having a plurality of controls for controlling the cleaning method; and controlling
the cleaning method via the control panel.

US-PAT-NO: 4545822

DOCUMENT-IDENTIFIER: US 4545822 A

TITLE: Process for cleaning vessels containing **sulfur** dichloride

---------- KWIC ---------

Process for cleaning vessels containing **sulfur** dichloride

Substantially all the liquid **sulfur** dichloride in a vessel is removed from the vessel leaving a heel (residue) of liquid and solid material on the bottom of the vessel. The remaining liquid is vaporized by heating the vessel, and the vapors are removed by vacuum and purging. The vessel is purged with an inert gas during heating to prevent exothermic reactions and the formation of toxic reaction products, and to assist in removal of vapors from the vessel. The removed liquid and vapors are neutralized by reaction with sodium hydroxide. The solid residue remaining in the vessel is then removed from both the bottom and wall portions.

The present invention is directed to an improved process for cleaning vessels containing **sulfur** dichloride. In the process of this invention, substantially all of the liquid **sulfur** dichloride is first removed from the vessel, and the material remaining in the vessel, including a slurry of liquid and solid material on the bottom, is heated in the presence of an inert gas to vaporize liquid components. The vapor is removed from the vessel by vacuum supplemented by the inert gas purge, neutralized, and the solid residue, primarily FeCl.sub.3, is then removed from the bottom of the vessel.

Sulfur dichloride, SCl.sub.2, is used extensively in the manufacture of organic chemicals and insecticides, as a vulcanizing agent for rubber, as a reagent, and as a lubricant additive. It is a brownish red liquid at room temperature which boils at about 60.degree. C. It spontaneously decomposes to form sulfur monochloride and chlorine at elevated temperatures.

Sulfur dichloride is conventionally prepared by the liquid phase chlorination of sulfur monochloride in the presence of a Lewis-acid catalyst such as FeCl.sub.3. Typical methods of preparation are disclosed in U.S. Pat. No. 3,219,413 to Kunkel et al. and U.S. Pat. No. 3,205,040 to Schmadebeck.

Sulfur dichloride is commercially shipped in liquid form in steel railroad tank cars. The sulfur dichloride is removed from the tank car through an opening in the top of the car leaving a mixture of liquid sulfur dichloride and solid material on the bottom of the tank car. This solid material is primarily ferric chloride with smaller amounts of ferrous chloride and ferrous sulfide. A residue of this solid material also coats the internal surfaces or walls of the tank car. Following normal shipping practice, only the sulfur dichloride is removed, and the tank car is then put back into service.

Vessels such as tank cars must be periodically static pressure-tested for leaks to meet regulatory requirements and to insure safe operating conditions. Prior to such testing, the vessel must be completely cleaned of reactive materials. Present cleaning techniques involve the addition of water or aqueous solutions of sodium hydroxide to the vessel. The water or sodium hydroxide solution reacts with the sulfur dichloride to form hydrochloric acid, chlorine, sulfur

and sulfur dioxide. These reaction products are, in general, less reactive and easier to handle than suflur dichloride. Hydrochloric acid, which is the primary reaction product, can be neutralized with additional sodium hydroxide and safely removed from the vessel.

Consequently, it is desirable to develop an improved process for cleaning vessels containing **sulfur** dichloride which is safer and more convenient than previous cleaning methods.

In accordance with the present invention, an improved process is provided for cleaning vessels containing **sulfur** dichloride. Substantially all of the liquid **sulfur** dichloride is first removed from the vessel leaving solid material wetted with **sulfur** dichloride on the bottom. The contents of the vessel are then heated in the presence of an inert gas to vaporize the liquid remaining in the vessel leaving a solid residue. The vapor is pumped from the vessel and the solid residue is finally removed from the bottom of the vessel. Heating the vessel can be conveniently accomplished by covering the vessel with a blanket of insulating material and blowing a stream of hot air against the side of the vessel.

The cleaning process of the present invention is broadly applicable to a wide variety of vessels which are suitable for containing **sulfur** dichloride. Such vessels are usually fabricated from steel and contain an inner lining or coating which forms from the reaction of **sulfur** dichloride with the steel. This inner coating which comprises primarily iron chlorides serves as a passivation layer preventing further corrosion of the internal steel surface.

While the present invention finds particular applicability

to railroad tank
cars which are the primary transportation mode for **sulfur** dichloride, other
vessels encompassed within the scope of this invention include storage tanks,
reactors and mixing equipment, tank trucks and ships. For the sake of
simplicity, however, specific features of this invention are described in
connection with railroad tank cars. This should not be construed as an intent
to limit the broad scope of the invention.

Prior to commencing the cleaning process of the present invention, it is first
necessary to remove as much of the free liquid **sulfur** dichloride as is
practicable from the bottom or heal of the tank car. This can be conveniently
accomplished by inserting a metal or plastic tube into the car to a point below
the liquid level, and applying a vacuum source to the other end of the tube.
The vacuum source must be sufficient to draw the liquid from the bottom of the
car into a receptacle. A suitable vacuum source for this purpose is a portable
vacuum truck. A receiving vessel to contain the liquid material and a scrubber
containing an aqueous solution of sodium hydroxide can be placed in the circuit
between the vacuum source and the tank car.

As liquid **sulfur** dichloride is drained from the car, it flows into a receiver.
The liquid **sulfur** dichloride, which can contain some dissolved **chlorine,** is
primarily isolated in the receiver. Vapors and droplets are conveyed under
vacuum to the sodium hydroxide solution in the scrubber. **Chlorine** and any
sulfur dichloride contained in the liquid are neutralized in the scrubber.

After removal of the free liquid **sulfur** dichloride on the bottom of the car,
residual liquid **sulfur** dichloride remaining in the car can

then be removed by vaporization. The residual **sulfur** dichloride is present as a surface film on the car walls and as a heel of liquid and solid material on the bottom of the car. This heel also contains a solid comprising primarily particles of ferric chloride, and a small amount of ferrous chloride, ferrous sulfide and other solid iron salts. These solids remain on the bottom and walls of the car after the vaporization of liquid material is complete.

Vaporization of the liquid **sulfur** dichloride remaining in the car is conveniently accomplished by heating the external surface of the car using a hot air blower system. The hot air blower can consist of one or more turbine heaters or a propane heater which directs a stream of hot air against the side of the tank car. In order to minimize heat loss and distribute heat evenly, the entire tank car can be covered with a sheet of insulating material such as a flexible fiberglass blanket. It is necessary to raise the internal temperature of the vessel to at least about 50.degree. C. and preferably to at least about 100.degree. C. to insure complete vaporization of the liquid in the vessel. Because the gaseous products evolved during vaporization are reactive in air, and a gas sweep can serve to facilitate vapor carry-over, a continuous stream of inert gas is supplied to the vessel. The inert gas is preferably nitrogen due to economic considerations, but in general any inert gas is suitable for this purpose. As an alternative heating procedure, the inert gas can be preheated to a temperature sufficient to effect the desired vaporization of liquid components.

1. A process for cleaning vessels containing liquid **sulfur** dichloride comprising the steps of:

(a) removing substantially all of the liquid **sulfur** dichloride from the vessel
leaving a solids-containing liquid slurry on the bottom of the vessel.

2. The process of claim 1 wherein the liquid **sulfur** dichloride is removed from
the vessel by vacuum.

3. The process of claim 2 wherein the liquid **sulfur** dichloride is neutralized
by reaction with sodium hydroxide.

US-PAT-NO: 5017240

DOCUMENT-IDENTIFIER: US 5017240 A

TITLE: Vapor treatment facilities for petroleum storage tank cleaning

---------- KWIC ---------

During storage of volatile petroleum distillate products such as gasoline, the mixture of air and gasoline vapors within the tank can contain up to about 35 percent hydrocarbons. Additionally, unrefined petroleum crude products such as untreated sour hydrocarbons, can contain sulfur compounds such as mercaptans, hydrogen sulphide, **sulfur dioxide** and the like. The discharge of the hydrocarbons or the sulfur compounds into the atmosphere during flushing of the tanks of increasing environmental concern. In some areas of the country, notably Southern California, existing or threatened legislation will prohibit the discharge of this vapor/air mixture directly into the atmosphere.

Referring now to FIG. 2, the treatment facility is illustrated in greater detail. As there illustrated, the bulk storage tank is evacuated with a conventional blower 14 having a capacity from 1000 to about 5000 cubic feet per minute and the air/vapor mixture is discharged into the first stage 34 of the treatment facility. This stage employs a caustic wash treatment in vessel 24 in which the air/vapor mixture is passed counter-current to a spray of caustic, typically aqueous solutions of about 30 weight percent sodium hydroxide to remove sulfur compound from the air/vapor mixture such as mercaptans, hydrogen

sulphate, **sulfur dioxide** and the like. The caustic solution is effective in removing the sulfur compounds which remain dissolved in the liquid and accumulate in the caustic wash tank 36. A liquid pump 38 removes the solution from the tank 36 and recycles it to a spray nozzle in the upper region of the vessel 34 through line 37.

US-PAT-NO: 4597803

DOCUMENT-IDENTIFIER: US 4597803 A

TITLE: Process for cleaning vessels containing **sulfur** dichloride

---------- KWIC ---------

Process for cleaning vessels containing **sulfur** dichloride

A process for cleaning vessels containing liquid **sulfur** dichloride is
disclosed. The **sulfur** dichloride is first removed from the vessel leaving a
heel (residue) of liquid and solid material on the bottom of the vessel. The
remaining liquid is vaporized by heating the vessel, and the vapors are removed
by vacuum and purging, and neutralized by reaction with a basic solution. The
vessel is purged with a dry inert gas during heating to prevent exothermic
reactions and the formation of toxic reaction products, and to assist in
removal of vapors from the vessel. The solid residue remaining in the vessel
is then removed from both the bottom and wall portions. In a preferred aspect,
the present invention is particularly applicable to cleaning railroad tank cars
which are used to transport **sulfur** dichloride.

The present invention is directed to an improved process for cleaning vessels
containing **sulfur** dichloride. In the process of this invention, substantially
all of the liquid **sulfur** dichloride is first removed from the vessel, and the
material remaining in the vessel, including a slurry of liquid and solid
material on the bottom, is heated in the presence of an inert gas to vaporize
liquid components. The vapor is removed from the vessel by

vacuum supplemented
by the inert gas purge, neutralized, and the solid residue, primarily
FeCl.sub.3, is then removed from the bottom of the vessel.

Sulfur dichloride, SCl.sub.2, is used extensively in the manufacture of organic
chemicals and insecticides, as a vulcanizing agent for rubber, as a reagent,
and as a lubricant additive. It is a brownish red liquid at room temperature
which boils at about 60.degree. C. It spontaneously decomposes to form sulfur
monochloride and chlorline at elevated temperatures.

Sulfur dichloride is conventionally prepared by the liquid phase chlorination
of sulfur monochloride in the presence of a Lewis-acid catalyst such as
FeCl.sub.3. Typical methods of preparation are disclosed in U.S. Pat. No.
3,219,413 to Kunkel et al. and U.S. Pat. No. 3,205,040 to Schmadebeck.

Sulfur dichloride is commercially shipped in liquid form in steel railroad tank
cars. The sulfur dichloride is removed from the tank car through an opening in
the top of the car leaving a mixture of liquid sulfur dichloride and solid
material on the bottom of the tank car. This solid material is primarily
ferric chloride with smaller amounts of ferrous chloride and ferrous sulfide.
A residue of this solid material also coats the internal surfaces or walls of
the tank car. Following normal shipping practice, only the sulfur dichloride
is removed, and the tank car is then put back into service.

Vessels such as tank cars must be periodically static pressuretested for leaks
to meet regulatory requirements and to insure safe operating conditions. Prior
to such testing, the vessel must be completely cleaned of reactive materials.
Present cleaning techniques involve the addition of water or aqueous solutions

of sodium hydroxide to the vessel. The water or sodium hydroxide solution reacts with the **sulfur** dichloride to form hydrochloric acid, **chlorine, sulfur and sulfur** dioxide. These reaction products are, in general, less reactive and easier to handle than **sulfur** dichloride. Hydrochloric acid, which is the primary reaction product, can be neutralized with additional sodium hydroxide and safely removed from the vessel.

Consequently, it is desirable to develop an improved process for cleaning vessels containing **sulfur** dichloride which is safer and more convenient than previous cleaning methods.

In accordance with the present invention, an improved process is provided for cleaning vessels containing **sulfur** dichloride. Substantially all of the liquid **sulfur** dichloride is first removed from the vessel leaving solid material wetted with **sulfur** dichloride on the bottom. The contents of the vessel are then heated in the presence of an inert gas to vaporize the liquid remaining in the vessel leaving a solid residue. The vapor is pumped from the vessel and neutralized by reaction with a base, and the solid residue is removed from the bottom of the vessel. Heating the vessel can be conveniently accomplished by covering the vessel with a blanket of insulating material and blowing a stream of hot air against the side or bottom of the vessel. Alternatively, atmospheric steam can be substituted for hot air, and polyethylene sheeting can be used as insulation.

The cleaning process of the present invention is broadly applicable to a wide variety of vessels which are suitable for containing **sulfur** dichloride. Such vessels are usually fabricated from steel and contain an

inner lining or
coating which forms from the reaction of **sulfur** dichloride with the steel.
This inner coating which comprises primarily iron chlorides serves as a
passivation layer preventing further corrosion of the internal steel surface.

While the present invention finds particular applicability to railroad tank
cars which are the primary transportation mode for **sulfur** dichloride, other
vessels encompassed within the scope of this invention include storage tanks,
reactors and mixing equipment, tank trucks and ships. For the sake of
simplicity, however, specific features of this invention are described in
connection with railroad tank cars. This should not be construed as an intent
to limit the broad scope of the invention.

Prior to commencing the cleaning process of the present invention, it is first
necessary to remove as much of the free liquid **sulfur** dichloride as is
practicable from the bottom or heel of the tank car. This can be conveniently
accomplished by inserting a metal or plastic tube into the car to a point below
the liquid level, and applying a vacuum source to the other end of the tube.
The vacuum source must be sufficient to draw the liquid from the bottom of the
car into a receptacle. A suitable vacuum source for this purpose is a portable
vacuum truck. A receiving vessel to contain the liquid material and a scrubber
containing a basic solution such as an aqueous solution of sodium hydroxide can
be placed in the circuit between the vacuum source and the tank car.

As liquid **sulfur** dichloride is drained from the car, it flows into a receiver.
The liquid **sulfur** dichloride, which can contain some dissolved **chlorine,** is
primarily isolated in the receiver. Vapors and droplets

are conveyed under vacuum to the scrubber. **Chlorine** and any **sulfur** dichloride contained in the liquid are neutralized in the scrubber by reaction with a suitable base such as sodium hydroxide, potassium hydroxide, calcium hydroxide, etc. The expression "neutralized" as used in this specification and appended claims is intended to denote the reaction of liquid or gaseous materials from the vessel with a basic material.

After removal of the free liquid **sulfur** dichloride on the bottom of the car, residual liquid **sulfur** dichloride remaining in the car can then be removed by vaporization. The residual **sulfur** dichloride is present as a surface film on the car walls and as a heel of liquid mixed with solid material on the bottom of the car. This heel also contains a solid comprising primarily particles of ferric chloride, and a small amount of ferrous chloride, ferrous sulfide and other solid iron salts. These solids remain on the bottom and walls of the car after the vaporization of liquid material is complete.

Vaporization of the liquid **sulfur** dichloride remaining in the car is conveniently accomplished by heating the external surface of the car using either a hot air blower system or atmospheric steam. The hot air blower can consist of one or more turbine heaters or a propane heater which directs a stream of hot air against the side of the tank car. Steam can be delivered through a pipe with openings directed at the bottom of the vessel. In order to minimize heat loss and distribute heat evenly, the entire tank car can be covered with a sheet of insulating material. A flexible fiberglass blanket can be used with a hot air system, or a polyethylene sheet can be used with steam.

1. A process for cleaning vessels containing liquid **sulfur** dichloride
comprising the steps of:

(a) removing substantially all of the liquid **sulfur** dichloride from the vessel
leaving a solids-containing liquid slurry on the bottom of the vessel,

2. The process of claim 1 wherein the liquid **sulfur** dichloride is removed from
the vessel by vacuum.

3. The process of claim 2 wherein the liquid **sulfur** dichloride is neutralized
after removal from the vessel.

4. The process of claim 3 wherein the liquid **sulfur** dichloride is neutralized
by reaction with sodium hydroxide.